

University of Groningen

Atomic force microscopy imaging of transition metal layered compounds

Kerssemakers, J. W. J.; de Hosson, J. T. M.

Published in:
Applied Physics Letters

DOI:
[10.1063/1.115440](https://doi.org/10.1063/1.115440)

IMPORTANT NOTE: You are advised to consult the publisher's version (publisher's PDF) if you wish to cite from it. Please check the document version below.

Document Version
Publisher's PDF, also known as Version of record

Publication date:
1995

[Link to publication in University of Groningen/UMCG research database](#)

Citation for published version (APA):

Kerssemakers, J. W. J., & de Hosson, J. T. M. (1995). Atomic force microscopy imaging of transition metal layered compounds: A two-dimensional stick-slip system. *Applied Physics Letters*, 67(3), 347-349.
<https://doi.org/10.1063/1.115440>

Copyright

Other than for strictly personal use, it is not permitted to download or to forward/distribute the text or part of it without the consent of the author(s) and/or copyright holder(s), unless the work is under an open content license (like Creative Commons).

The publication may also be distributed here under the terms of Article 25fa of the Dutch Copyright Act, indicated by the "Taverne" license. More information can be found on the University of Groningen website: <https://www.rug.nl/library/open-access/self-archiving-pure/taverne-amendment>.

Take-down policy

If you believe that this document breaches copyright please contact us providing details, and we will remove access to the work immediately and investigate your claim.

Downloaded from the University of Groningen/UMCG research database (Pure): <http://www.rug.nl/research/portal>. For technical reasons the number of authors shown on this cover page is limited to 10 maximum.

Atomic force microscopy imaging of transition metal layered compounds: A two-dimensional stick-slip system

J. Kerssemakers, and J. Th. M. De Hosson

Citation: *Appl. Phys. Lett.* **67**, 347 (1995); doi: 10.1063/1.115440

View online: <https://doi.org/10.1063/1.115440>

View Table of Contents: <http://aip.scitation.org/toc/apl/67/3>

Published by the [American Institute of Physics](#)

Articles you may be interested in

[Exact rate calculations by trajectory parallelization and tilting](#)

The Journal of Chemical Physics **131**, 044120 (2009); 10.1063/1.3180821

[Lateral stiffness: A new nanomechanical measurement for the determination of shear strengths with friction force microscopy](#)

Applied Physics Letters **70**, 1548 (1997); 10.1063/1.118639

[Accurate quantum calculation for the benchmark reaction \$\text{H}_2 + \text{OH} \rightarrow \text{H}_2\text{O} + \text{H}\$ in five-dimensional space: Reaction probabilities for \$J=0\$](#)

The Journal of Chemical Physics **99**, 5615 (1993); 10.1063/1.465954

[Summary Abstract: Fabrication of ultrathin heterostructures with van der Waals epitaxy](#)

Journal of Vacuum Science & Technology B: Microelectronics Processing and Phenomena **3**, 724 (1985); 10.1116/1.583125

[Lateral vibration effects in atomic-scale friction](#)

Applied Physics Letters **104**, 083103 (2014); 10.1063/1.4866427

[WSXM: A software for scanning probe microscopy and a tool for nanotechnology](#)

Review of Scientific Instruments **78**, 013705 (2007); 10.1063/1.2432410



Measure Ready
155 Precision I/V Source

A new current & voltage source
optimized for scientific research

LEARN MORE ▶

Atomic force microscopy imaging of transition metal layered compounds: A two-dimensional stick-slip system

J. Kerssemakers and J. Th. M. De Hosson^{a)}

Department of Applied Physics, University of Groningen, Nijenborgh 4, 9747 AG Groningen, The Netherlands

(Received 28 October 1994; accepted for publication 19 May 1995)

Various layered transition metal dichalcogenides were scanned with an optical-lever atomic force microscope (AFM). The microscopic images indicate the occurrence of strong lateral stick-slip effects. In this letter, two models are presented to describe the observations due to stick-slip, i.e., either as a static or as a dynamic phenomenon. Although both models describe correctly the observed shapes of the unit cell, details in the observed and simulated images point at dynamic nonequilibrium effects. This exact shape of the unit cell depends on cantilever stiffness, scan direction, and detector direction. © 1995 American Institute of Physics.

Layered transition metal dichalcogenides exhibit some remarkable two-dimensional behavior, despite their three-dimensional atomic structure. The crystallographic structure can be described as a hexagonal close-packed layer of transition metal atoms, sandwiched in between two layers of chalcogen atoms (S, Te, Se) with the same symmetry. Unit cells MX_2 , in which M represents the transition metal and X a chalcogen, are connected by relatively strong chemical bonds within the sandwich but only weakly bonded to adjacent sandwiches. Because of this particular atomic arrangement the physical properties exhibit a rather strong anisotropic behavior.

In the past, extensive scanning tunneling microscopy (STM) and atomic force microscopy (AFM) studies on these materials have been carried out. Observed effects are charge density waves (CDW)^{1,2} and pinning of CDW by point defects by STM^{3,4} as well as periodic lattice distortions (PLD) by AFM.⁵ However, observations of nonperiodic features at an atomic scale with AFM are rather scarcely available as compared to observations of the atomic structure as such. The latter can easily be observed in various types of layered compounds, even when the estimated tip-contact area and contact force are far beyond atomic dimensions. Therefore, the observed corrugations in those cases must be a collective effect of many individual atomic interactions involving high stress and strain components.

This letter proposes a simple physical model describing the essence of the mechanisms involved when imaging various layered transition metal compounds with AFM. In contrast to earlier stick-slip studies, attention will not be focused merely on the occurrence of stick-slip. The novel message is to show that the detailed shape of the unit cell can be ascribed to a two-dimensional stick-slip behavior of the AFM tip provided dynamical effects are taken into account as well.

Imaging was performed with an optical-level Nanoscope II AFM. Although the current version is not implemented for measurement of the lateral force component, due to the non-zero tip height, the observed force variation will always con-

sist of a mixture of forces perpendicular and parallel to the substrate.⁶

Various materials, such as TiS_2 and $NbSe_2$, were scanned in air, water, and ethanol, at scan speeds ranging from 3 to 15 000 nm/s. There was no qualitative difference observed, although scanning in liquid always resulted in a much better defined and more stable experiment. In water and with tip loads less than 0.1 nN, single particles of 0.3 nm were clearly visible at an atomic resolution. With a gradual increase of the applied force, the force corrugation increases while single features (edges, particles) disappear or are smeared out because of the increase of the contact area. In the range between 10 and 50 nN (substrate degradation) a typical back and forward scan line along a high-symmetry lattice direction in TiS_2 is depicted in Fig. 1. The characteristic features are the following: Along the main directions of the hexagonal lattice, a regular sawtooth is observed. Next, inversion symmetry upon scanning exists instead of a mere shift, as can be deduced from either starting slopes. This feature implies that the main origin of the cantilever torsion is due to lateral effects. Finally, independent of hysteresis loop size, the corresponding slopes in back and forward scan lines are aligned. This still holds when the lattice shows modulation, as in the case of ReS_2 .

Since the loop does not depend on the scan size or the scan velocity piezohysteresis is not very probable. Degradation effects (loose flakes) are also unlikely, as the effect is also observed on freshly cleaved materials which cannot be

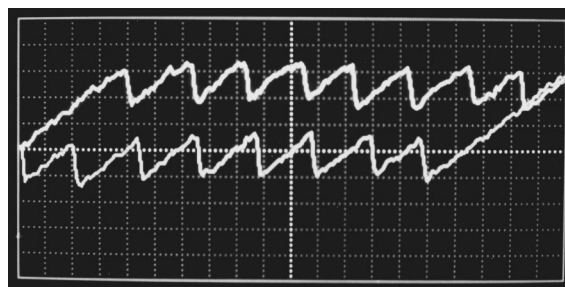


FIG. 1. An AFM scan on $NbSe_2$ along a $[1000]$ direction. Forward (lower) and backward (upper) scan signal. This kind of scan signal produces images similar to Fig. 3. The observed sawtooth periodicity corresponds to the lattice distance.

^{a)}Electronic mail: hossonj@phys.rug.nl

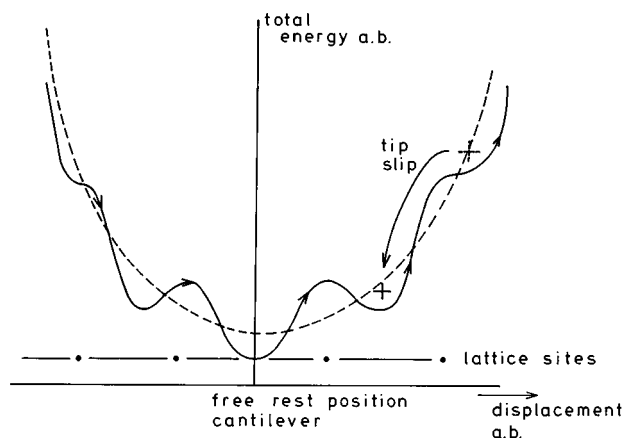


FIG. 2. Simplified energy concept of a stick-slip process. Plus sign: tip position; dashed line: energy tip vs displacement; solid line with arrows: energy scanning tip with lattice interaction vs displacement.

degraded at all within the current force range (up to ~ 1000 nN).

The characteristic features are all typical properties of a stick-slip system. In Fig. 2, a simple one-dimensional model is presented. A sinusoidal potential is moved continuously with respect to a fixed tip position. The other side of the spring is connected to a mass, from which the movement relative to the fixed position can be monitored. The total energy versus displacement will show a minimum up to a certain displacement at which the second derivative (stiffness) will disappear. The system itself will run along with a certain minimum up to this point, which represents the stick phase. Then it will start accelerating towards a zero displacement, i.e., the slip phase. The amount of damping determines whether enough energy will be dissipated by the system to come to a final stop in the next minimum. Back and forward scans will be y-axis symmetric in this picture, causing the hysteresis loop as well as the stick-phase alignment.

In this physical concept, “ideal” or geometric stick-slip will only occur when the minimum of the lattice potential show singularities in the first derivative. Acceleration as well as deceleration become infinite in the next minimum of the total energy versus displacement curve. In practice, this can dynamically only be generated by a system that combines a low mass, a strong tip-lattice interaction, and a strong damping. In that case the corresponding signal will be perfectly sawtoothlike, with only rapidly decaying oscillations after a particular slip.

In two dimensions, the nature of geometrical as well of dynamical stick-slip will be more complex. In the geometrical case, one may think of discrete hexagonal lattice sites to which the system either sticks completely or slips instantaneously. In a dynamical system, the movement of the tip mass will be determined to some extent by the system equilibrium points. Just as in the one-dimensional case, the momentary dynamical state can be affected by the system properties like low mass, strong tip-sample interaction, and damping.

To investigate the physics behind these observations two different approaches to the stick-slip behavior are investigated, i.e., a geometrical and a dynamical model, respec-

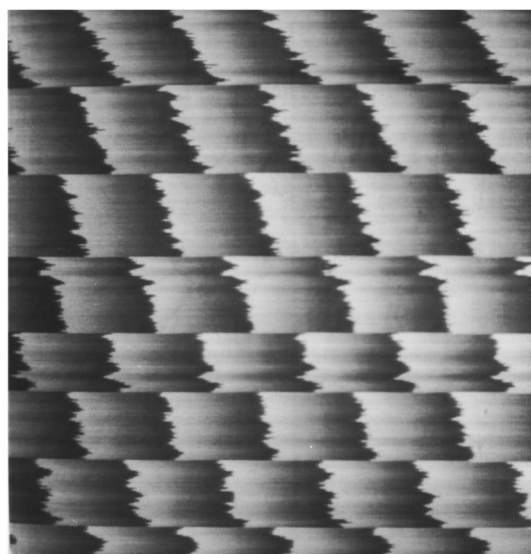


FIG. 3. An AFM image of NbSe₂ along the [1000]-scan direction; scan-size 2.2 nm. The detector is aligned along [1000] as well, which means that a lateral movement along this direction will produce a maximal signal amplitude.

tively. Both mimic the scan movement along a hexagonal lattice in any chosen configuration. The geometrical model contains all the essential physics in the slip criterion. Here, a simple criterion of an isotropic threshold displacement is assumed. The system then slips to the site which gives a maximal relaxation of tip displacement, which turns out to be dependent on scan angle and threshold displacement. In contrast, the dynamical model employs a two-dimensional sinusoidal potential and a damping term proportional to the tip velocity. Position, velocity, and acceleration are calculated in first order from the total energy at a particular tip displacement. To mimic the slip behavior correctly it turns out that of the order of 100 first-order system state iterations per image pixel are necessary. The ratio between damping and velocity is chosen to be constant.

In this study on chalcogenides, numerous AFM images are compared to the model predictions. Figure 3 displays an experimental AFM image of NbSe₂ along a $[-1\ 2\ -1\ 0]$ direction with parallel detector alignment, followed by the predictions based on the geometrical model in Fig. 4 and the results of the dynamical model in Fig. 5. A large slip threshold in the geometric model and a large lattice-potential-to-spring constant ratio in the dynamic model had to be incorporated. Both imply a strong tip-sample interaction, although the good resemblance to the geometric model suggests nearly ideal stick-slip as well.

High-load images in all scan configurations (order 100 nN and beyond) are well described by these models. Some details in the actual image (oscillations and certain onsets of the scans) can only be described by the dynamic model.

When the scans are made at a high applied force and closely (i.e., a few degrees off) along a principal hexagonal direction of the lattice, either model predicts the tip slip along the main direction with an occasional single or multiple (zigzag) side jump. The latter strongly depends on the spring constant and the lattice potential. With a proper detec-

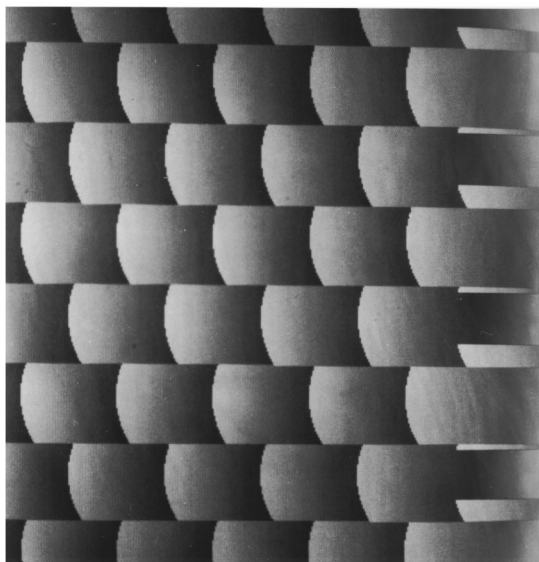


FIG. 4. An AFM image that a tip produces when forced to follow a continuous $[1000]$ -scan path. The tip is allowed to slip and stick to discrete sites. Scan and detector configuration are identical to that of Fig. 3.

for these leaps aside may cause a much stronger detector signal than slip along the main direction. In such a case, a single or a multiple slip aside show up as ripples, both in real and calculated images. The periodicity of the ripples is determined by the relative scan angle and is not only predicted but also correctly measured up to a rather macroscopic scale (Fig. 6).

In conclusion we may state that the assumptions of stick-slip movement of the AFM-tip holds even in the high-load regime. The geometrical model turns out to be an as-

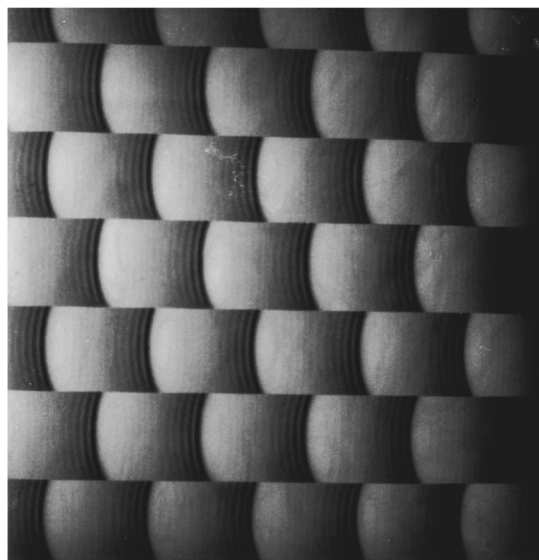


FIG. 5. An AFM image produced by a mass-spring system that scans along a $[1000]$ direction of a sinusoidal potential. Scan and detector configuration are identical to that of Fig. 3. Note the oscillations after each slip.

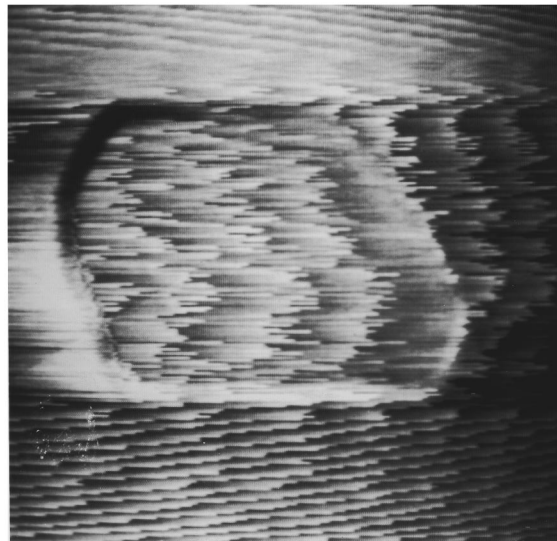


FIG. 6. An AFM image of NbSe_2 , scan size is 328 nm. Ripples with periods much larger than atomic distances are observed at scans close (within 1°) to a $[1000]$ -direction. These ripples are ascribed to leaps-aside of the tip at a low frequency. The large rectangular area is made by scanning with a high load prior to taking the present image.

ymptotic approximation of a dynamical model. In practice it is a nearly ideal stick-slip regime. This cannot be explained using a single spring and single potential model. Together with the high stress involved (up to ~ 10 GPa) it suggests at least a significant load-stiffening deformation around the tip-sample interface. The occurrence of strong nanoscale deformations in these stick-slip systems is also supported by the fact that the occurrence of stick-slip is not affected by the actual shape of the tip in a qualitative sense at least, but depends rather on the one- or multilayered surface structure itself.⁷⁻⁹

The work described in this letter is part of the research program of the Foundation for Fundamental Research on Matter (FOM-Utrecht) and has been made possible by financial support from the Netherlands Foundation for Technical Sciences (STW-Utrecht).

¹R. V. Coleman, B. Giambattista, P. K. Hansma, A. Johnson, W. W. McNairy, and C. G. Slough, *Adv. Phys.* **37**, 559 (1988).

²C. G. Slough, W. W. McNairy, R. V. Coleman, J. Garnaes, C. B. Prater, and P. K. Hansma, *Phys. Rev. B* **42**, 9255 (1990).

³G. P. E. M. van Bakel, J. Th. M. De Hosson, and T. Hibma, *Appl. Phys. Lett.* **56**, 2402 (1990).

⁴G. P. E. M. van Bakel and J. Th. M. De Hosson, *Phys. Rev. B* **46**, 2001 (1992).

⁵Y. Gong, Q. Xue, Z. Dai, C. G. Slough, R. V. Coleman, and L. M. Falicov, *Phys. Rev. Lett.* **71**, 3303 (1993).

⁶S. J. O'Shea, M. E. Welland, and T. M. H. Wong, *Ultramicroscopy* **52**, 55 (1993).

⁷R. M. Overney, H. Takano, M. Fujihira, W. Paulus, and H. Ringsdorf, *Phys. Rev. Lett.* **72**, 3546 (1994).

⁸S. Lee, H. Miyazaki, S. D. Mahanti, and S. A. Solin, *Phys. Rev. Lett.* **62**, 3066 (1989).

⁹D. Tomanek, G. Overney, H. Miyazaki, and S. D. Mahanti, *Phys. Rev. Lett.* **63**, 877 (1989).